Microelectronics Journal 41 (2010) 860–864

Contents lists available at ScienceDirect

Microelectronics Journal

journal homepage: www.elsevier.com/locate/mejo

Integrated CNTs thin film for MEMS mechanical sensors

Van Thanh Dau [a,*], Takeo Yamada [b], Dzung Viet Dao [a], Bui Thanh Tung [a], Kenji Hata [b], Susumu Sugiyama [a]

[a] Graduated School of Science and Engineering, Ritsumeikan University, 1-1-1 Higashi, Kusatsu, Shiga, 525-8577, Japan
[b] Nanotube Research Center, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba Center 5, 1-1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan

ARTICLE INFO

Article history:
Received 2 February 2010
Accepted 20 July 2010
Available online 24 August 2010

Keywords:
MEMS process
EB
CNTs film
SWNT
Super growth
Piezoresistive effect
Gauge factor

ABSTRACT

This paper reports the top-down fabrication of CNTs thin film on MEMS structure, and characterization of piezoresistive coefficients of aligned single wall carbon nanotube (SWNT) forest film. The film was synthesized by water-assisted chemical vapor deposition (CVD), a process known as "super growth". CNTs film was condensed, manually maneuvered and conveniently patterned by EB lithography to form desirable shapes. The longitudinal and transverse gauge factors of the CNTs thin film were measured to be 3.75 and 0.67, respectively.

1. Introduction

Research on carbon nanotubes (CNTs) for sensing application is a very promising direction for nanoscale devices due to CNTs' excellent electrical and mechanical properties. Besides the characteristics of either metallic conductor or semiconductor which allows CNTs to become promising components of integrated circuit to continue shrink the transistor size, CNTs posses a tensile strength larger than any other known materials [1]. CNTs have been used in various applications for tunable electrometrical oscillators, chemical and physical sensors, non-volatile memory, actuator, field-emitting flat panel displays and also nanotube radio.

The integration of CNTs in MEMS is usually relies either on the bottom-up (in situ growth) or the top-down (post-growth) methods. The bottom-up technique utilized catalytic particles directly patterned onto a substrate to control the position of CNTs, while the top-down method focused on manipulation of the growth CNTs into their desired position. The bottom-up, self-assembly has been known as a pattern to assemble nanotubes; Li. et al. [2] have reported on aligned network of self-assembly carbon nanotube by Langmuir–Blodgett technique and Kang et al. [3] have also successfully fabricated multilayer systems by substrate oriented growth technique. Although these methods particularly have advantages in the area of field-effect transistor, the complexity and control of the assembly are the main obstacles for MEMS integration and functional devices.

Alternative top-down methods to realize integrated micro/nano device system are to contact nanotubes into the patterned structure. In order to accurately deposit the nanotube at the desired location, several methods have been investigated. Based on the principle that synthesized SWNTs were preferentially attracted to the ($-NH_2$) functionalized surface, Liu et al. [4] reported on the surface functionalization technique. This can be useful for positioning nanotube, but has very poor directional control as the nanotubes often loop around the patterns. Several applications of this method to position nanotubes in between pairs of electrodes have been reported and only limited rate of reliable electrical contact of the device can be assured [5,6]. Huang et al. reported on fluidic manipulation, utilizing flow assembly on a functionalized substrate. With a number of control techniques, this method can achieve parallel array of nanotube and even crossbar structure by single or by alternating the flow in orthogonal directions [7,8]. Another, rather fast, self-assembly method is the dielectrophoretic (DEP) manipulation in which CNTs were trapped and aligned by either or both AC/DC fields. This is the most promising method due to its precise manipulation in non-contact manner, although it is vital to ensure the length of nanotube and to have appropriate electrode size, geometry as well as applied voltage [9–12]. The final top-down solution reported in the literatures is nanorobotic manipulation, directly pick the CNTs by atomic force microscope (AFM) or transmission electron microscope (TEM) and place them at the desired place, or to contact them after random deposition on a substrate with electron beam lithography. The system can operate with sub-nanometer positioning resolution [13,14] and the

* Corresponding author. Tel.: +81 77 5615097; fax: +81 77 5613994.
E-mail address: van@fc.ritsumei.ac.jp (V.T. Dau).

0026-2692/$ - see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.mejo.2010.07.012

nanotubes can be bended, twisted, slided, rolled and broken [15–19]. Obviously, this method is suitable for investigating fundamental properties rather than for large-scale fabrication.

In order to be effectively applied to MEMS devices, CNTs should have well-controlled properties, orientation, be easily integrated to the system. An alternative and more realistic is to assemble a massive quantity of CNTs at desired locations and form it into well-defined configuration by conventional top-down lithography. Recent approach has been reported by Hayamizu et al. [20]. This process allows complex CNT component as a fundamental shape-engineerable unit for integrated device system and, therefore, potentially opens a way for low-cost smart CNTs-based MEMS devices.

One of the main interests of the CNTs properties is the piezoresistive effect. The effect has been investigated for the first time by Tombler et al. [21]. A gauge factor (the CNTs sensitivity to strain) of 1000, i.e. five times larger than that of single crystal silicon, was reported. Other measurements of Cao et al. [22] and Stampfer et al. [23] showed the gauge factor up to 3000 for pre-strained individual SWNTs. For practical application, Zhao et al. [24] have shown the potentials of SWNT as strain sensor by embedding CNTs in a polymer to measure the stress applied into test specimen. Further study was presented by Li et al. [25] using thin film of nanotubes as strain sensor. The film was made of randomly orientated bundles of SWNTs and was attached to a rubber strip using epoxy. Using the DEP method, Sickert et al. [26] have presented a macro-scale strain sensing device with bundle single-walled carbon nanotube. Another concept with multi-walled CNTs film was reported recently by Li et al. [27] and Song et al. [28]. Piezoresistive behavior of CNTs has also been applied for pressure sensor [29,30] and force sensor [31]. Although the gauge factor was found to be rather small for CNTs films, impressive tensile strength with elongation over 50% [32] allows the material to be applied in large deformation sensing device.

This paper reports on the integration of CNTs film to the MEMS structure and the characterization of piezoresistive coefficients of integrated CNTs element. The test specimen was made of aligned single wall carbon nanotube (SWNT) forest films, which are possible by the top-down process. The film was synthesized by water-assisted chemical vapor deposition (CVD), and then, it is condensed, manually maneuvered and conveniently patterned by EB lithography to form desirable shapes. The advantages of the method are fast alignment, array design, mass production, high level structural diversity and complexity. Remarkably, due to parallel process ability, the specimens were tested on either longitudinal or transverse direction of applied stresses; thus, both longitudinal and transverse piezoresistive coefficients of CNTs film were measured at the same time.

2. Integration of CNT film to microstructure

Recently, CNTs have been synthesized by water-assisted chemical vapor deposition (CVD), a process known as "super growth". The "super growth" CNTs are of high purity, and as long as millimeter scale (Fig. 1a). Because of its large scale, conventional MEMS technique can be used to integrate CNTs to microstructure as illustrated in Fig. 1. Self-assembled SWNTs were vertically aligned in form of sparse forest films and then zipped into high-density SWNT films by liquid-induced zippering effect. After positioning the film onto the prefabricated silicon device, lithography is performed to pattern the film into desired shapes.

Specifically, a $500 \times 500 \times 4\ \mu m^3$ ($L \times W \times T$) CNT film was firstly synthesized by water-assisted chemical vapor deposition. Very long and catalyst-free SWNTs with diameter of 2.8 nm had carbon purity higher than 99.9%, were vertically aligned and occupied 3–4% of the total volume of this forest film [33]. 0.3 μm Cr/Au electrodes were created by a lift-off process on a silicon wafer with passivation layer (Si_3N_4) on top (Fig. 1(i–ii)). Thanks to the large size of the forest film, CNT film is manually placed on electrodes within isopropyl alcohol (IPA) solution and naturally dried. Surface tension of the solution and strong van der Waals interaction accordantly compress CNT film into a 0.3 μm thick layer and form a strong contact with electrodes (Fig. 2(iii)). Once densified, the SWNTs film could be considered as a continuous material layer. Hydrogen silsesquioxane (HSQ) resist was spin-coated and baked at 90 °C for 10 min. HSQ was then patterned by electron beam (EB) lithography and developed by tetra-methylammoniumhydroxide (TMAH) solution (2.38%, ZTMA-100, Zeon) (Fig. 2(iv)). Then, CNT film was etched by reactive ion etching (RIE) (RIE-200L, Samco) with oxygen plasma and argon to define the designed shape (Fig. 2(v)). The HSQ mask was finally removed by buffered hydrofluoric acid (HF) (Fig. 2(vi)).

Generally, the densification of the CNTs forest film happened in all directions; however the interaction of the substrate and the forest together with longitudinal SWNT orientation drive the densification in only one direction and essentially pull the films toward the substrate with strong adhesion, resulting in flat and cohesive CNT surface, on which we could uniformly coat HSQ

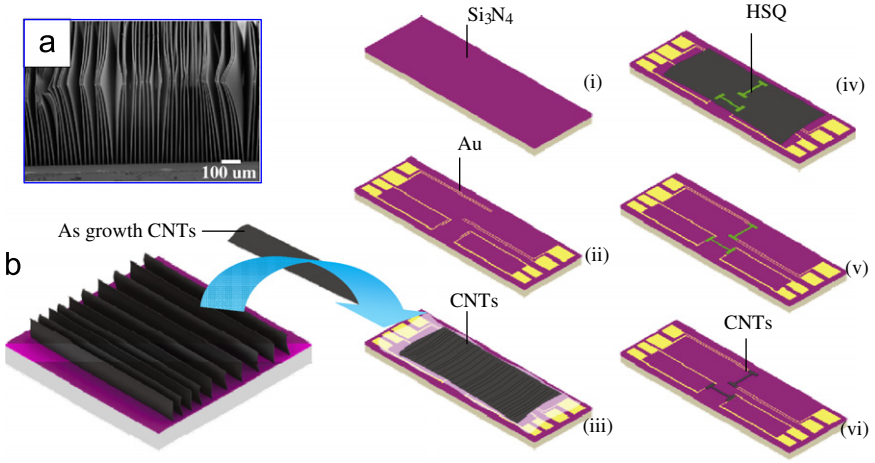

Fig. 1. Fabrication process of test device. Super-growth synthesized carbon nanotube (a, [33]) will be patterned on silicon structure by EB lithography. CNT film is put on Cr/Au electrode in IPA solution (iii) and CNT element formed by O_2 plasma etching (v).